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SOME CLAY-WATER PROPERTIES OF CERTAIN CLAY MINERALS

BY

R. E. GRIM AND F. L. CUTHBERT

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SOME CLAY-WATER PROPERTIES OF CERTAIN CLAY MINERALS

BY

R. E. Grim and F. L. Cuthbert†

ABSTRACT

Computed values are given for the thickness of the water film adsorbed on the surfaces of the various clay minerals when clays composed of such minerals develop specific plastic characteristics. Based on such values, the following concept of clay-water relationships is presented: The dominant factor determining the plastic properties of clays is the rigidity of the water held on the surfaces of the clay minerals, and the point of beginning of the transition of completely rigid water to liquid water is marked by great changes in such properties. Each type of clay mineral seems to have a characteristic ability to stabilize water, and the exchangeable ions also exert an influence. The reaction between water and kaolinite or halloysite may require considerable time so that there is frequently a time lag after mixing clays composed of such minerals before the plastic properties are fully developed.
Applications of the foregoing concept in the fields of geology, ceramics, and soil mechanics are suggested.

I. Introduction

In the course of an investigation of the constitution and bonding and related properties of clays for metallurgical use under the sponsorship of the Illinois State Geological Survey, the Department of Mechanical Engineering of the University of Illinois, and the Illinois Clay Products Company, Joliet, Illinois, a fundamental study was made of the bonding characteristics (as shown in molding sands) of types of clay based on their clay-mineral composition. In addition to information regarding the bonding properties of clays, the study has provided some data on general clay-water relationships that seem to have broad applications in industry, engineering, and science. It is the purpose of this paper to present these findings regarding the general clay-water relationship together with the experimental data that support them and a discussion of their application. The detailed results of the study as they pertain to bonding clays and molding sands are being published elsewhere.[1]

II. Thickness of Water with a Definite Configuration Adsorbed on Basal Plane Surfaces of Montmorillonite

When the compression strength is determined for rammed specimens of sand-clay mixtures that contain various amounts of tempering water, results similar to those shown in Fig. 1 are obtained. In such curves, the tempering water is shown in percentage of total weight of the sand-clay mixture. Figure 1, *(A)* and *(B)*, shows a group of curves for a series of mixtures with varying amounts *(A)* of sodium-montmorillonite clay and *(B)* of calcium-montmorillonite clay. The data on which these clay-mineral identifications are based are being published elsewhere.[1] It is obvious from the curves that the maximum strength of the sand-clay mixture or the maximum bonding power of the clay is developed for each mixture when there is a precise and narrowly limited amount of tempering water present.

Table I shows the thickness of the water on each unit flake of montmorillonite when there is just enough water to produce the maximum green strength shown in Fig. 1. These computations are based on the assumption that all basal plane surfaces of
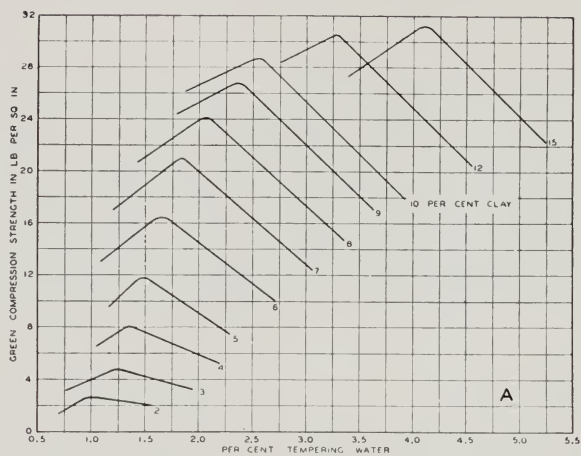
Fig. 1 (*A*).—Relation of compression strength to tempering water for sand-clay mixtures containing various amounts of sodium-montmorillonite clay.
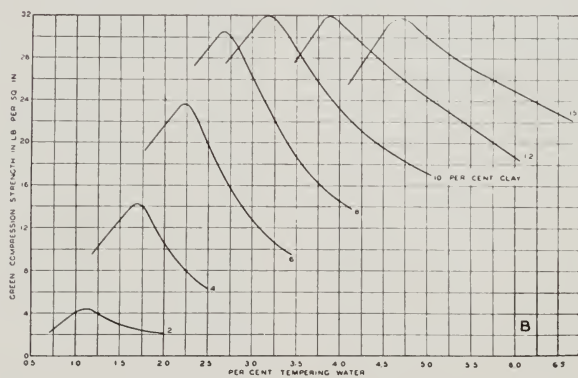

Fig. 1 (*B*).—Relation of compression strength to tempering water for sand-clay mixtures containing various amounts of calcium-montmorillonite clay.
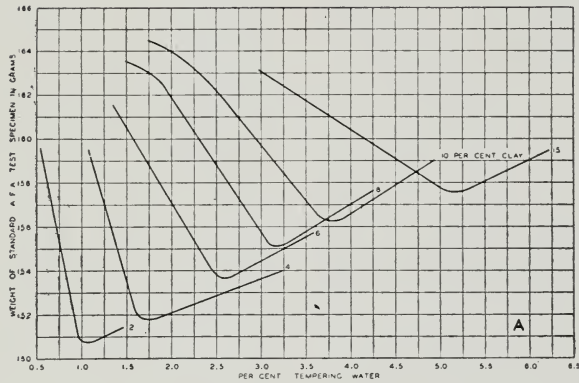
FIG. 2 (*A*).—Relation of bulk density to tempering water for sand-clay mixtures containing various amounts of sodium-montmorillonite clay.
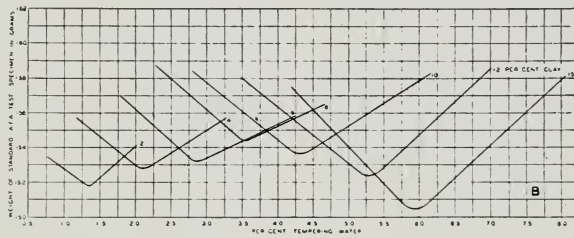

FIG. 2 (*B*).—Relation of bulk density to tempering water for sand-clay mixtures containing various amounts of calcium-montmorillonite clay.

Clay in mixture (%)	Sodium-montmorillonite clay			Calcium-montmorillonite clay		
	Dry (110°C.) wt. of clay (gm.)*	Water (cc.)*	Water-film thickness in molecular layers†	Dry (110°C.) wt. of clay (gm.)*	Water (cc.)*	Water film thickness in molecular layers†
6	111	33	3	111	45	4
7	129	37	3			
8	147	42	3	147	54	4
9	156	48	3			
10	184	52	3	184	64	3+
12	222	66	3	222	77	3+
15	277	83	3	277	94	3+

*These values are based on a 2000-gm. mixture; dry weight (110°C.) of clay is less than the percentage value because of water loss from room temperature to 110°C.

†A single layer of water one molecule thick per unit cell of montmorillonite is equal to 0.1 gm. of water per gm. of clay if it is assumed that the water has the loose packing required by the configuration of Hendricks and Jefferson (footnote 2(a)).

units of montmorillonite are available to the penetration of water and the development of water layers. This assumption is valid since it is well known that montmorillonite tends to break down in the presence of water to unit cell size.

The computations show the striking result that, regardless of the amount of clay, the maximum bonding strength is developed in the sodium-montmorillonite clay when there is just enough water to coat each basal surface with a water layer three molecules thick.

Recently it has been rather well established that the water[2] held on the basal surfaces of flakes of montmorillonite is made up of water molecules that tend to be arranged in a definite pattern. The water is, therefore, in a rigid condition rather than fluid. The configuration of the water molecules is determined largely by the arrangement of oxygen atoms in the surface layers of the montmorillonite, and, as a consequence,

[2](a) S. B. Hendricks and M. E. Jefferson, "Structures of Kaolin and Talc-Pyrophyllite Hydrates and Their Bearing on Water Sorption of Clays," *Amer. Mineralogist,* **23** [12] 863–75 (1938); *Ceram. Abs.,* **22** [4] 113 (1939).

(b) R. E. Grim, "Modern Concepts of Clay Materials," *Jour. Geol.* **50** [3] 225–75 (1942); *Ill. Geol. Survey Rept. Inves.* No. 80 (1942); *Ceram. Abs.,* **21** [8] 177 (1942).

should extend to a limited distance from the montmorillonite surface.

The concept has been developed elsewhere[1] that maximum green compression strength in sand-clay-water mixtures develops when the clay-water component has the maximum thickness and maximum degree of rigidity for a given clay content. If this be true and molding-sand characteristics provide a considerable body of supporting evidence, then the tempering water of maximum green compression strength is a measure of the maximum amount of water that can be held with the water molecules in about perfect orientation. The sodium montmorillonite retains about completely oriented water molecules to a thickness of 3 molecules (about 9 a.u.) on each basal unit surface and calcium montmorillonite to a thickness of 4 molecules (about 12 a.u.) on each basal unit surface.

If the bulk density is determined for a sand-clay-water mixture that has been rammed in a mold and if such bulk density values are plotted against variations in amount of tempering water, curves such as those shown in Fig. 2, *(A)* and *(B),* will be obtained. These figures show that for each proportion of sand and clay there is a precise, and narrowly limited, amount of water wherein minimum bulk density is developed. Minimum bulk density develops at that moisture content which gives the mixture the greatest resistance to packing during the ramming in a mold.[1]

Table II gives computed values of the thickness of the layer of water on each unit flake of montmorillonite when there is just enough water to permit the greatest resistance to packing, that is, at the point of minimum bulk density. Again it is assumed that each basal plane surface of montmorillonite is available to water penetration. The computation shows that for the sodium montmorillonite this condition is reached when water is present equal to a layer 4 molecules thick (about 12 a.u.) and for the calcium montmorillonite when water is present equal to a layer 5 molecules thick (about 15 a.u.). In other words, for each of these two montmorillonite clays an extra

TABLE II.—WATER-FILM THICKNESS PER UNIT CELL OF MONTMORILLONITE AT MINIMUM BULK DENSITY

Clay in mixture (%)	Sodium-montmorillonite clay			Calcium-montmorillonite clay		
	Dry (110°C.) wt. of clay (gm.)*	Water (cc.)*	Water-film thickness in molecular layers	Dry (110°C.) wt. of clay (gm.)*	Water (cc.)*	Water film thickness in molecular layers
6	111	52	4+	111	57	5
8	147	63	4+	147	71	5
10	184	76	4	184	86	5—
12				222	106	5—
15	277	103	4—	277	119	4+

*Values based on 2000-gm. mixture.

molecular water layer above those that are held with maximum rigidity is necessary to develop maximum resistance to packing.

It seems logical that maximum resistance to packing would prevail not when the clay water coating of the quartz grains is completely rigid but when there is a certain adherence between the coatings of the grains, such as would occur in the presence of some incompletely rigid (but not liquid) water. It seems likely, therefore, that the addition of a single layer of water above that amount held completely rigid provides water in which the water molecules are not perfectly oriented. That is, beyond a layer 3 water molecules thick for sodium montmorillonite, and 4 water molecules thick for calcium montmorillonite, the perfection of the orientation of the water molecules is reduced but not destroyed completely. It seems clear that the perfection of orientation of the water molecules adsorbed by these montmorillonites decreases gradually rather than abruptly for thicknesses greater than 3 or 4 molecules, respectively.

The data indicate that the thickness of completely rigid water is greater when calcium rather than sodium is the exchangeable base. This does not mean that the total thickness of water that can be held with any degree of orientation of the water molecules is greater for calcium than for sodium. In fact, the well-known thixotropic properties of water suspensions of sodium-montmorillonite clays and other data from molding sand studies suggest that the reverse is true.

The reason for the thicker coating of completely rigid water when calcium is the chief exchangeable base is not definitely known, but it is probably related to the fact that the calcium ion hydrates whereas the sodium ion does not.[3] The exchangeable ions are held close to or at the surface of the montmorillonite flakes, and the hydration of the calcium ion might well be an additional force tending to develop configuration in the water molecules (the main force being the orientation of the oxygen atoms in the basal surface of the montmorillonite itself). Other factors, such as the force tending to hold the montmorillonite flakes together,[2(b)] probably largely control the total thickness of water that can develop with any definite configuration, so it does not follow from this reasoning that, when calcium ions are present, the water layer with any degree of configuration of the molecules should be thickest.

There is no basis for computing the thickness of the adsorbed water film in clays composed of other clay minerals because water does not penetrate readily between each individual layer. As shown in Figs. 3, 4, and 5, curves for halloysite, kaolinite, and illite clays, representing the relation of tempering water to green compression strength and to minimum bulk density, have the same general shape as those for montmorillonite clays. From this fact and other considerations of the bonding properties of these clays (for example air-set strength, see section IV), it seems certain that water in which there is a definite configuration of the molecules develops on the available plane surfaces of these clay minerals.

III. CHARACTER OF ABSORBED WATER IN RELATION TO PLASTIC PROPERTIES

If the thickness of the water held by the clay minerals in a completely rigid condition is the important factor controlling the bond-

[3]S. B. Hendricks, R. A. Nelson, and L. T. Alexander, "Hydration Mechanism of the Clay Mineral Montmorillonite Saturated with Various Cations," Jour. Amer. Chem Soc., 62 [6] 1457-64 (1940); Ceram. Abs., 19 [11] 265 (1940).
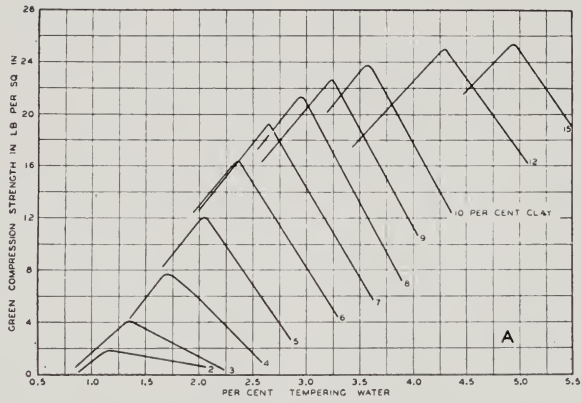
Fig. 3 (*A*).—Sand-clay mixtures containing halloysite clay showing relation of compression strength to tempering water.
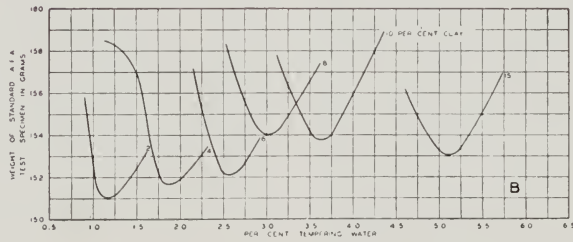

Fig. 3 (*B*).—Sand-clay mixtures containing halloysite clay showing relation of tempering water to bulk density.

Fig. 4 (*A*).—Sand-clay mixtures containing kaolinite clay showing relation of compression strength to tempering water.

Fig. 4 (*B*).—Sand-clay mixtures containing kaolinite clay showing relation of tempering water to bulk density.

Fig. 5 (*A*).—Sand-clay mixtures containing illite clay showing relation of compression strength to tempering water.

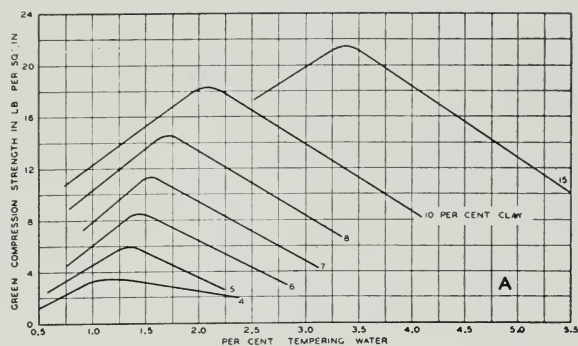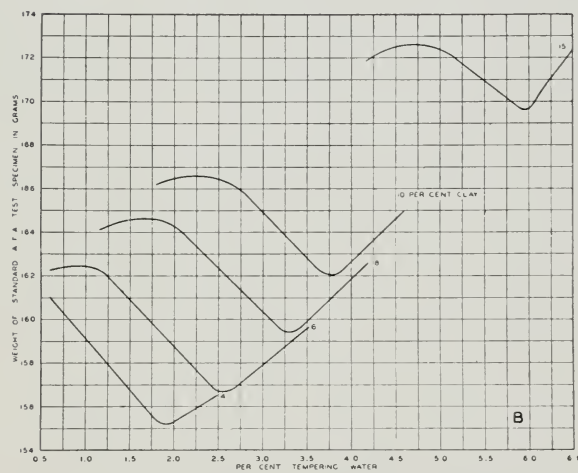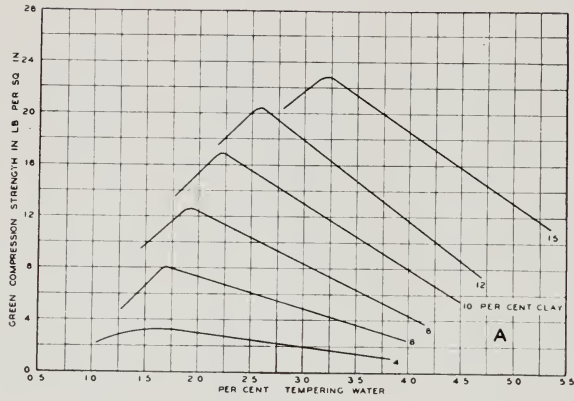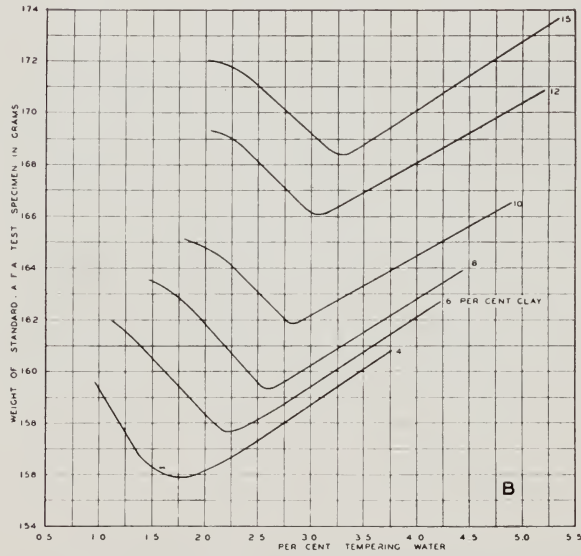Fig. 5 (*B*).—Sand-clay mixtures containing illite clay showing relation of tempering water to bulk density.

TABLE III.—WATER CONTENT IN PERCENTAGE DRY (100°C.) WEIGHT OF CLAY AT MAXIMUM GREEN COMPRESSION STRENGTH AND MINIMUM BULK DENSITY

Clay in sand-clay mixtures (%)	Illite clay		Kaolinite clay	
	Max. green compression strength	Min. bulk density	Max. green compression strength	Min. bulk density
8	24	32	22	42
10	23	29	22	40
12	22	26		
15	22	23	33	41

ing strength of clays in molding sands, it would seem that it should also be of importance in controlling other plastic properties of clays. In this connection, the following data and discussion are offered:

Some years ago, a careful study[4] was made on a small laboratory extrusion machine of the relation between the power necessary to extrude various kinds of clay and their water content. In Fig. 6, curves showing some of the results of this work are presented. Curve *A* is for a kaolinite clay, *B* and *C* for clays composed of both kaolinite and illite, and *D, E,* and *F,* for clays composed entirely of illite. The outstanding feature of the curves is that with more

[4]R. E. Grim and R. A. Rowland, unpublished studies made in laboratories of the Illinois State Geological Survey, Urbana, Ill.

water than a certain minimum amount, extrusion requires very little power. With water decreasing below this minimum amount, the power necessary to extrude increases rapidly to the point where extrusion is impossible. In other words, there is a very narrow moisture content that marks the transition from easy extrusion to impossible extrusion. An examination of Table III and Fig. 6 shows that maximum green compression strength and minimum bulk density develop for kaolinite and illite clays in the moisture range that is the critical transition range in the extrusion curves. The conclusion seems warranted therefore that in the extrusion of clays, extrusion is about impossible as long as there is no more water than that which will be fixed rigidly by the clay minerals. When the amount of water is increased above this point so that water with incomplete rigidity is present, extrusion becomes easy.

Further, it would seem justifiable to suggest that the presence of water with a definite configuration and the point of the beginning of the transition of such water to liquid water are important, perhaps dominant, factors underlying the plastic properties of clays in general. The foregoing data give a first quantitative measure

TABLE IV.—AIR-SET STRENGTH OF RAMMED SAND-CLAY MIXTURES

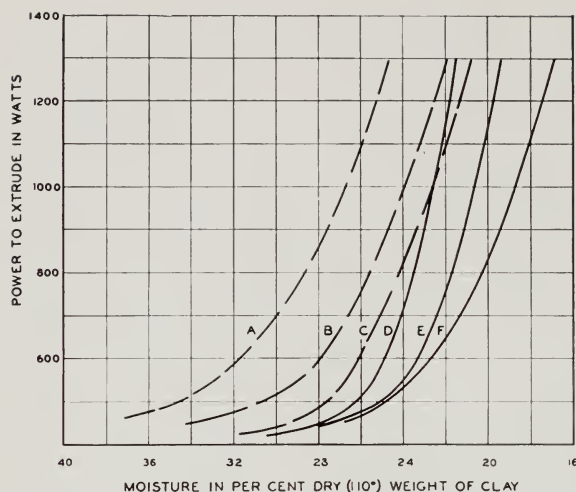| Green compression strength determined immediately after ramming (lb./sq. in.)		Compression strength determined the following periods of time after ramming (lb./sq. in.)							
		15 min.		30 min.		1 hr.		3 hr.	
Strength	Moisture	Strength	Moisture	Strength	Moisture	Strength	Moisture	Strength	Moisture
Halloysite clay (12% clay mixture)									
7.0	4.75	14.9	4.45	20.8	4.3	31.5	3.82	51.0	2.45
3.0	5.95	4.0	5.7	5.2	5.4	9.5	5.1	52.5	3.95
Kaolinite clay (10% mixture)									
7.6	3.8	13.2	3.35	17.2	2.95	21.8	2.55	30.0	1.50
4.0	5.2	5.5	4.75	8.0	4.40	13.5	3.7	40.0	1.95
Illite clay (12% clay mixture)									
8.5	4.2	12.2	3.65	15.4	3.37	20.4	2.9	32.3	2.05
5.05	5.4	8.5	4.85	11.6	4.47	17.0	3.95	33.3	2.70
Montmorillonite clay (6% mixture)									
7.75	3.2	9.90	2.85	11.0	2.66	15.8	2.3	27.6	1.70
5.87	4.6	7.0	4.3	8.8	4.0	11.3	3.70	22.8	2.9

FIG. 6.—Curves showing relation of tempering water in clays and the power necessary to extrude clays through an auger machine.

of the transition point in the physical state of the water adsorbed by clays.

IV. TIME FACTOR IN CLAY-WATER RELATIONS

Investigations of the bonding strength of various classes of clays have shown that when rammed specimens of mixtures of sand and halloysite clay or kaolinite clay are allowed to stand in air for short periods of time there is a gradual increase in the bonding strength of the clay without much loss of water (air-set strength). As shown in Table IV, sands bonded with montmorillonite clays and illite clays show no appreciable gain in strength without an accompanying loss of water.

The explanation for air-set strength seems to be that a certain amount of time is required for the water to penetrate some of the basal plane surfaces of the cells of halloysite and kaolinite and to assume fixed positions. Immediately after mixing halloysite-clay-sands with water they have a "wet" feel which disappears within a short time (1 hour ±) without an accompanying loss of water. Montmorillonite-clay-sands show a complete development of strength immediately without air-set strength because water

penetrates at once to all available surfaces of montmorillonite. Illite does not break down into individual cells in the presence of water and there is little penetration of water to individual basal surfaces in such clays, and as a consequence there is no air-set strength.

The air-set property shows that for certain clay minerals there is an appreciable amount of time required for water to penetrate to available basal plane surfaces and to become fixed. Under similar conditions for other clay minerals, the penetration is almost instantaneous, and for still others there is no penetration. There is, therefore, a time factor that must be considered in measuring the plastic properties of clays composed of certain clay minerals.

V. APPLICATIONS OF SUGGESTED CLAY-WATER RELATIONSHIPS

The clay-water characteristics suggested herein seem to have wide applications in explaining the origin and geologic history as well as the properties of clay materials. Thus, they should help to understand the processes of diagenesis and the difference in the texture between many sediments composed of illite and those composed of kaolin-

ite; sediments composed of the former clay mineral tend to have a laminated texture. They would enter into an understanding of the structures that sometimes develop on a large scale in sediments that are largely unconsolidated.

In the field of ceramics, the range of water contents yielding plastic clay bodies probably corresponds to the water content in excess of that held in a completely rigid condition and somewhere below the amount where fluid water is abundant. The difficulty in removing water during drying appears to be much greater for oriented rigid water than for fluid water.

In the field of engineering, particularly in soil mechanics, the hitherto puzzling absence of variations of water content with depth encountered in certain soils and clays may find an explanation. Further, it would seem that the clay-water relationship presented herein should explain some of the changes taking place in soils under superimposed load.

VI. Summary

The rigidity of the water held on the surfaces of the clay minerals seems to be a dominant factor determining the bonding and plastic properties of clays. The point of the beginning of the transition of completely rigid water to liquid water is marked by great changes in such properties.

Each type of clay mineral seems to have a characteristic ability to stabilize water, and the exchangeable ions also exert an influence.

In the clay minerals halloysite and kaolinite, the reaction with water is not completed immediately, so that there may be a considerable time lag after mixing the clay and water before certain plastic or bonding properties develop.

The foregoing clay-water characteristics seem to have applications in industry, engineering, and science. Brief illustrations of such applications are given for the fields of geology, ceramics, and soil mechanics.